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(54) **Separation of zeolite particles from colloidal dispersions thereof**

(57) A method of separating zeolite crystal particles from a liquid colloidal dispersion thereof comprises initially cooling the dispersion for example with CO_2 snow, to form a slush and effecting filtration as the slush melts. In this way, it is possible to employ conventional filtration apparatus, such as a vacuum filter, to obtain a filter cake of the zeolite crystal particles which could not be obtained by direct filtration of the liquid dispersion.

Certain of the chemical formula(e) appearing in the printed specification were submitted in formal form after the date of filing.

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SPECIFICATION

Separation of zeolite particles from colloidal dispersions thereof

This invention relates to a novel method of separating colloidal particles from an aqueous solution, and particularly relates to separating fine zeolite crystals from their mother liquor.

Crystalline aluminosilicate zeolites, such as ZSM-5 and ZSM-36, may be prepared from an aqueous mother liquor solution containing sources of alkali metal oxide, aluminum oxide, silicon oxide, and ethylenediamine, pyrrolidine or tetrapropylammonium cations (U.S. Patents 3,702,886 and 4,016,245). The crystals produced are generally in the size range of 0.005 to 0.3 micron in diameter and remain suspended or dispersed in the mother liquor in a colloidal state (U.S. Patents 3,992,466 and 3,926,782). Since ordinary filters have paper or canvas filter cloths with pore sizes of approximately 40 microns, such filters are impractical for use in separating the zeolite particles from the mother liquor.

In attempting to solve the problem of separating the dispersed zeolites from the solution, the prior art has suggested the use of various agglomerating agents, centrifuges, flocculants and frothing systems (U.S. Patent 3,902,993). A simple, yet highly effective method has now been developed which is quite suitable for laboratory or commercial processes and has the added advantage in that the method does not require the use of costly additional equipment.

The present invention relates to a process for separating colloidal-sized zeolite crystals from a liquid dispersion of the particles, such as would be encountered in the crystalline growth of the zeolite from an aqueous solution. In accordance with the invention, the liquid disperson or mother liquor containing the crystals is cooled to form a slush. The slush is allowed to melt and the colloidal particles or crystals are filtered from the melting slush with conventional filter apparatus.

In a preferred embodiment of the invention, the cooling of the liquid dispersion is accomplished by mixing or adding CO_2 snow to the liquid. The snow converts the liquid to a creamy slush which may be filtered with conventional filter apparatus to obtain a zeolite filter cake and a clear filtrate.

The following Example illustrates the invention.

Approximately 30 pounds of ZSm-5-type zeolite crystals were produced in a 100-gallon container by procedures similar to those outlined in the literature. The crystals were approximately 0.3 micron in diameter, and all attempts at separation of the crystals from the solution with regular filtration techniques and standard filtration cloths failed, since the zeolite passed through the filter cloth with the filtrate.

The dispersion was frozen to a creamy slush by the addition of CO_2 snow. CO_2 snow is a crystalline precipitant of carbon dioxide and may be formed by allowing liquid carbon dioxide from a pressurized cylinder to expand to atmospheric pressure. CO_2 snow in an amount equal to approximately 25 weight percent of the liquid dispersion will result in the desired slush consistency.

The slush was transferred to a vacuum filter having an ordinary canvas membrane and allowed to melt. Filtration proceeded during the course of melting to obtain a clear filtrate and zeolite filter cake. Although a vacuum filter is preferred, the melting slush may be satisfactorily filtered with non-vacuum apparatus.

CLAIMS

1. A method of separating zeolite particles from a liquid colloidal dispersion of the particles, which comprises cooling the liquid disperson of the particles to form a slush, allowing the slush to melt and filtering the melting slush to obtain a residue of zeolite particles.

2. A method according to Claim 1, wherein said cooling is accomplished by the addition of CO_2 snow to said liquid dispersion of the particles.

3. A method according to Claim 2, wherein the CO_2 snow is added to the liquid dispersion in an amount equal to approximately 25 weight percent of the liquid dispersion.

4. A method according to Claim 1, 2 or 3, wherein the melting slush is filtered with a vacuum filter to obtain a clear filtrate and a zeolite crystal filter cake.

5. A method according to Claim 1, 2, 3 or 4, wherein the zeolite is a ZSM-5-type zeolite.

6. Zeolite particles whenever obtained by the method claimed in any preceding claim.

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